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The Wood-Witt Program is a shorthand title given to a group of coordinated efforts designed to identify and eliminate undesirable defects and impurities in GaN. It was begun by Cohn Wood (ONR) and Jerry Witt (AFOSR) in October of 1999. A few groups were directly funded to work on this task but many others also choose to participate because of the chance to compare results on well characterized samples. Presently, in September of 2002, some 37 people, from 7 different countries, have either supplied material or made measurements, and the total number of participants is at least 79.

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Final

~~Annual~~ Technical Report

to

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Introduction

The Wood-Witt Program is a shorthand title given to a group of coordinated efforts designed to identify and eliminate undesirable defects and impurities in GaN. It was begun by Colin Wood (ONR) and Jerry Witt (AFOSR) in October of 1999, and the original goal was to reduce donor N_D and acceptor N_A concentrations in state-of-the-art GaN by a factor ten, from the low-10^{17} to the low-10^{16} cm^{-3} level. A few groups were directly funded to work on this task, but many others also choose to participate because of the chance to compare results on well characterized samples. Presently, in September of 2002, some 37 people, from 7 different countries, have either supplied material or made measurements, and the total number of participants is at least 79, as listed in Appendix I. The first samples investigated were mainly hydride vapor phase epitaxial (HVPE) GaN layers grown on Al_2O_3 by Richard Molnar of Lincoln Laboratory/MIT. These layers had thicknesses of up to 70 μm, and world-record 300-K mobilities, at the time, of > 950 cm^2/V-s in the thickest samples. Within a two-year period, this material was thoroughly characterized by many different groups, and quite well understood. For example, it was shown that the donor and acceptor concentrations in the thick samples were $N_D \cong 1 \times 10^{17}$ cm^{-3} and $N_A \cong 3 \times 10^{16}$ cm^{-3} [1]. However, it was found that the GaN/Al_2O_3 interface regions had much higher concentrations, with $N_D \cong 1 \times 10^{20}$ cm^{-3} and $N_A \cong 5 \times 10^{19}$ cm^{-3} [1-3]. It was further shown that the donors in the bulk region were mostly Si, and in the interface region, O, and that the dominant acceptor was the Ga vacancy (V_{Ga}), everywhere [3]. Measurements on about 100 different HVPE layers, obtained from many independent sources, revealed a highly conductive (degenerate) interface layer in every single case, and the cause was a diffusion of the shallow donor O from the Al_2O_3 into the GaN. This O diffusion is concentrated in a region of about 2000 Å in the Molnar case, and this same region has a high density of threading dislocations; thus, the O may move into the GaN by pipe diffusion along the dislocations. This phenomenon should be studied further in the next phase of the W-W program, and micro-SIMS measurements should help solve this problem. In any case, the existence of a thin, highly conductive interface layer has a strong effect on electrical and optical properties, and must be considered in the data analysis [2].

In 2001, HVPE GaN material from the Samsung Advanced Institute of Technology (SAIT) became available for the W-W program when AFOSR, in conjunction with their Tokyo arm, AOARD, was able to strike a deal with SAIT. The Samsung GaN wafers had first been grown on Al_2O_3, to a thickness of about 500 μm, then separated from their Al_2O_3 substrates, and finally ground, etched and polished to a thickness of

about 200 µm [4]. Since the GaN was separated from the Al_2O_3, experiments could be conducted on both the Ga and N faces. The material was excellent, in fact meeting the original goals of the W-W program. So far, the lowest donor (shallow) and acceptor concentrations measured are: $N_D = 7 \times 10^{15}$ cm^{-3}, and $N_A = 1.3 \times 10^{15}$ cm^{-3} [5]. The highest 300-K mobility is 1320 cm^2/V-s, and the highest peak mobility is close to 12000 cm^2/V-s. Donor-bound exciton photoluminescence (PL) linewidths are less than 0.5 meV [6], and both O and Si donors have been identified by PL. In contrast to the Molnar GaN, the dominant donor in the bulk seems to be O, not Si, but the dominant acceptor is still the Ga vacancy. Partly because of the low free-carrier concentrations, Schottky barriers are excellent, having barrier heights of 1.27 eV on the Ga face, and 0.75 eV on the N face [7]. Furthermore, the Samsung GaN is very useful for electron-irradiation experiments, because of the small background concentrations of donors, acceptors and traps. It also has a small threading dislocation density, $< 1 \times 10^6$ cm^{-2}, at the top (Ga) face. This means that the average distance between dislocations is about 10 µm, so that it should be possible to study the Cottrell atmosphere of impurities and defects surrounding each dislocation. The micro-SIMS capability at Ohio State University (Prof. Brillson) should be very useful in this investigation. Overall, many experiments have been performed on the relatively small amount of material received from Samsung, and 35% of the publications listed in Appendix II are related to these studies. It is likely that Samsung GaN wafers will have a significant impact on the quest to develop a GaN substrate for homoepitaxy, and it is noteworthy that the most detailed characterization studies on these wafers have come out of the W-W program.

Electron irradiation for defect studies

To create point defects, in our laboratory, we use high-energy (0.7-2.0 MeV) electrons from a van de Graaff accelerator. Also, lower energy electrons (0.3 – 1.0 MeV) are available from a similar but smaller machine at Hanscom AFB. Most of the energy loss in high-energy electron bombardment occurs from electron-electron, rather than electron-nucleus, collisions. Such e-e collisions limit the electron range in GaN to about 0.7 mm for 1-MeV electrons, for example. For epitaxial layers, of thickness 100 µm or less, the electrons lose very little energy in traversing the sample. If a relativistic electron of energy E makes a *direct* hit on a nucleus, it will transfer a maximum energy E_m given by

$$E_m = \frac{2E(E + 2m_e c^2)}{Mc^2} = \frac{2.147 \times 10^{-9} E(E + 1.022 \times 10^6)}{A} \qquad (1)$$

where m_e and M are the electron and ion masses, respectively, A is the atomic weight, and the energies are in eV. The threshold energy E_{th} necessary to produce an atomic displacement is then just given by the condition $E_m = E_d$, where E_d is the displacement energy. In GaN, Van Vechten [8] has estimated E_d values of 32.5 and 24.3 for N and Ga displacements, respectively, and it can be shown from Eq. 1 that these values lead to threshold energies E = 0.18 and 0.51 MeV, for production of V_N-N_I and V_{Ga}-Ga_I Frenkel pairs, respectively.

Temperature-dependent Hall-effect (T-Hall)

T-Hall measurements constitute the standard method of determining donor N_D and acceptor N_A concentrations in semiconductor materials [9]. Ideally, the temperature dependences of both the Hall mobility μ_H and the carrier concentration n (assumed n-type) are fitted to determine N_D, N_A, and E_D, the donor activation energy. The mobility, for elastic scattering processes, can be calculated from $\mu_H = e\langle\tau^2\rangle/m^*\langle\tau\rangle$, where $\langle\tau^n\rangle$ denotes an average of the nth power of the relaxation time $\tau(E)$ over electron energy E. The relaxation *rate* $\tau^{-1}(E)$ has contributions from various scattering mechanisms:

$$\tau^{-1}(E) = \tau_{ac}^{-1}(E) + \tau_{pe}^{-1}(E) + \tau_{po}^{-1}(E) + \tau_{ii}^{-1}(E) + \tau_{dis}^{-1}(E) \tag{2}$$

in which acoustical-mode lattice vibrations scatter electrons through the deformation potential (τ_{ac}) and piezoelectric potential (τ_{pe}); optical-mode vibrations through the polar potential (τ_{po}); ionized impurities and defects through the screened coulomb potential (τ_{ii}); and charged dislocations, also through the coulomb potential (τ_{dis}). The strengths of these various scattering mechanisms depend upon certain lattice parameters, such as dielectric constants and deformation potentials, and extrinsic factors, such as donor, acceptor, and dislocation concentrations, N_D, N_A, and N_{dis}, respectively. For thick, HVPE GaN layers, N_{dis} is usually not important, and the only fitting parameter is N_A, since the ionized defect/impurity density is given by $N_I = 2N_A + n \approx 2N_A + n_H$, where n_H is measured in the experiment. In reality, since polar-optical scattering is not elastic, we often use a more accurate fitting scheme for μ_H vs. T [9].

To determine N_D and E_D we must solve the charge-balance equation (CBE):

$$n + N_A = \frac{N_D}{1 + n/\phi_D} \tag{3}$$

where $\phi_D = (g_0/g_1)N_C'\exp(\alpha_D/k)T^{3/2}\exp(-E_{D0}/kT)$. Here, g_0/g_1 is a degeneracy factor (= ½ for an s-state), N_C'

$= 2(2\pi m_n^* k)^{3/2}/h^3$, where h is Planck's constant, E_D is the donor energy, k is Boltzmann's constant, and E_{D0} and α_D are defined by $E_D = E_{D0} - \alpha_D T$. If more than one donor exists within a few kT of the Fermi energy, then equivalent terms, involving N_{D2}, N_{D3}, etc., are added on the right-hand side of Eq. 3.

Recently we have applied the above analyses to μ_H vs. T (Fig. 1) and n_H vs. T (Fig. 2) data for a very pure free-standing HVPE GaN layer grown by Samsung [10]. The 300-K and peak mobilities of this sample were 1245 and 7400 cm^2/V-s, respectively, and the fitted donor and acceptor concentrations were 6.7 x 10^{15} and 1.7 x 10^{15} cm^{-3}, respectively. Interestingly, this value of N_A is very close to the V_{Ga} concentration measured by PAS in similar material [11]. In fact, V_{Ga} is often the dominant acceptor in undoped HVPE GaN, over a wide range of acceptor concentrations [3]. After 1-MeV electron irradiation, both N_D and N_A increase, each by about 1 cm^{-3} for each bombarding electron per cm^2, giving a production rate of about 1 cm^{-1} [1]. From various considerations, it has been argued that the donor is likely an N vacancy V_N, and the acceptor an N interstitial N_I [1]. Also determined from the experiment is the donor activation energy $E_D \approx$ 0.06 eV. This value is compatible with the theoretical conclusion that V_N should be a shallow donor [12]. Also, theory predicts N_I to have a deep-acceptor state [12], consistent with the Hall data. If we are indeed seeing only N-sublattice damage, then the absence of Ga-sublattice damage is a mystery.

Deep level transient spectroscopy (DLTS)

DLTS is a technique capable of determining electron and hole trap parameters: concentration, activation energy, and capture cross section [9]. In its common form, a reverse-biased Schottky barrier or p-n junction is subjected to a forward-bias pulse in order to flood the depletion region with electrons (or holes), and thus temporarily fill the traps in that region. Upon returning to the original reverse bias, the temporarily trapped electrons or holes will be re-emitted, as illustrated in Fig. 3. For experiments involving a Schottky barrier on n-type material, there will be a capacitance C immediately before the pulse, and C - ΔC immediately afterwards. In first order, the trap concentration N_T is given by $N_T = -2N_D(\Delta C/C)$, where N_D is the net shallow donor concentration. As time proceeds after the filling pulse, the capacitance will return to its original value C, usually in an exponential manner. In the box-car technique, this exponential is sampled at two times, t_1 and t_2, to effect a "rate window" defined by $r = \ln(t_2/t_1)/(t_2-t_1)$. As temperature is swept upwards, the emission rate e_n of a particular trap increases, according to $e_n = CT^2\exp(-E/kT)$, where C is a constant involving the capture cross section, and E is an activation energy, consisting of the trap energy E_T

plus the cross-section barrier height E_σ, if non-zero (i.e., $E = E_T + E_\sigma$). As illustrated in Fig. 4, the DLTS "spectrum" then consists of a series of peaks due to different traps N_{Ti}, with each peak occurring at the temperature for which $e_{ni} = r$.

Fig. 1. Mobility fit for Samsung freestanding GaN layer.

Fitting parameters

$E_1 = 13.5$ eV (if $c_L = 382$ GPa)

$P_\perp = 0.083$ (or $h_{pz} = 0.49$ C/m^2)
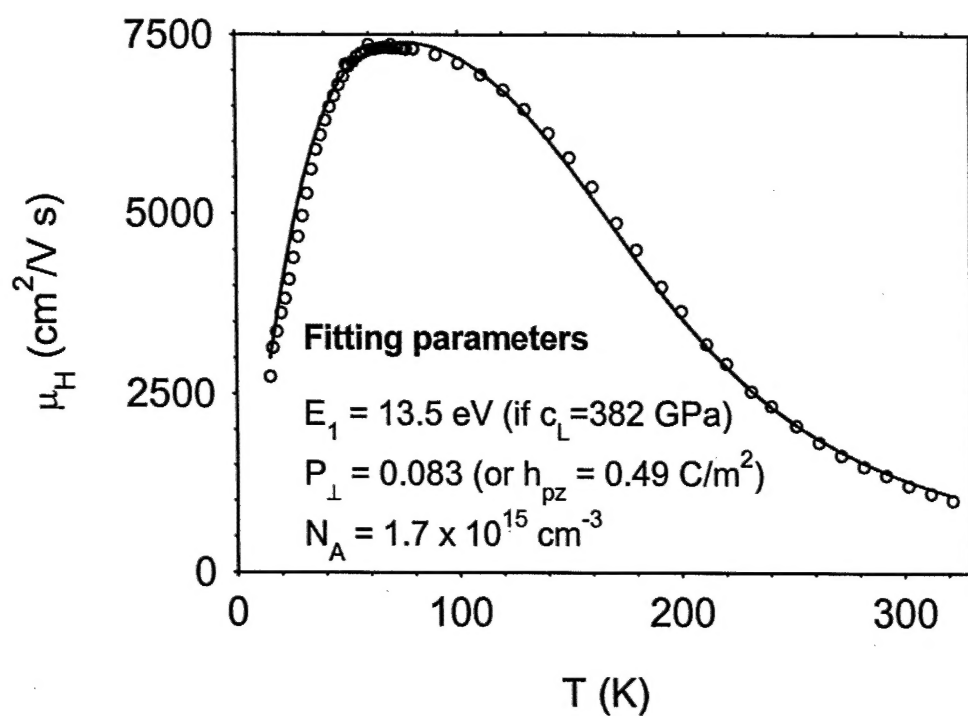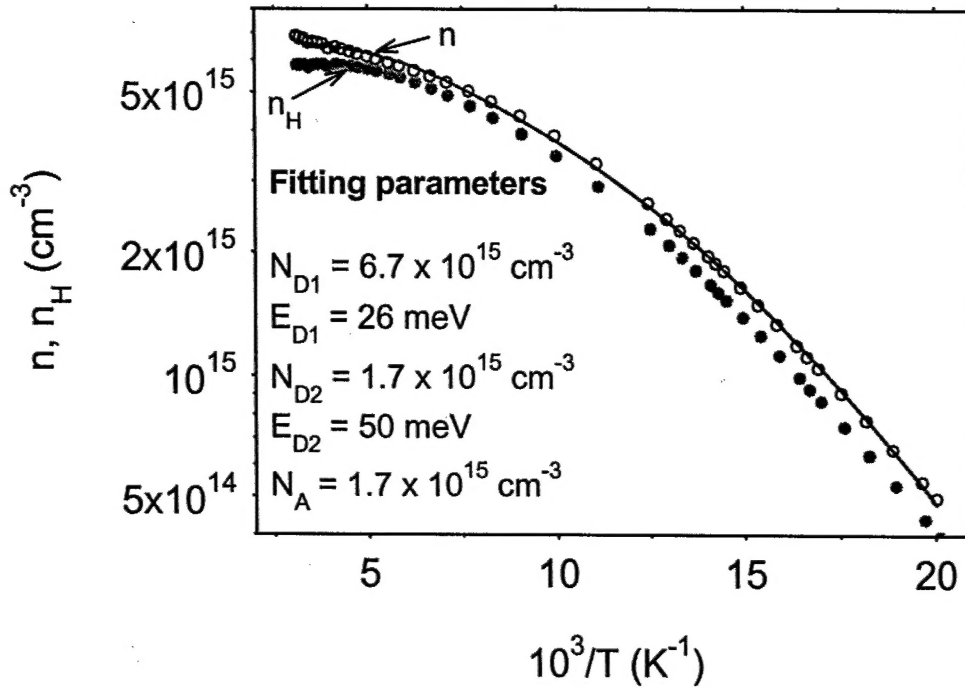
$N_A = 1.7 \times 10^{15}$ cm^{-3}

Fig. 2. Carrier concentration fit for Samsung freestanding GaN layer: n_H is the Hall concentration and n is the true concentration.

Actually, the simple formula, $N_T = -2N_D(\Delta C/C)$, holds only if $N_T/N_D \ll 1$, and if E_T is not very large. We expect these conditions to be often violated in AlGaN. Fortunately, we have derived a general formula, contained in a recent article on DLTS [13] written for the *Encyclopedia of Materials: Science and Technology*. It can be shown that a precise form of $\Delta C/C$ is

$$-\frac{\Delta C}{C} = \frac{\left[1 + f_\lambda \dfrac{N_T(w_r - \lambda)}{N_D{}^{net}(w_r)}\right]^{1/2} - 1}{\left[1 + f_\lambda \dfrac{N_T(w_r - \lambda)}{N_D{}^{net}(w_r)}\right]^{1/2}} \tag{4}$$

where

$$w_r = \sqrt{\frac{2\varepsilon\left(\phi_B - V_r - E_{C\infty}/e - kT/e\right)}{eN_D^{net}}} \tag{5}$$

and

$$\lambda = \left(\frac{2\varepsilon(E_T - E_{C\infty} - kT)}{e^2 N_D^{net}}\right)^{1/2} \tag{6}$$

The positions of w_r and λ are shown in Fig. 3. The most important part of Eq. 4 is a unique expression for f_λ that is valid for both high trap concentrations and deep trap energies [14]:

$$f_\lambda = \frac{1}{\left[1 + \frac{\alpha\beta^{1/2}}{(1+\alpha-\alpha\beta)^{1/2}}\right]^2}\left\{F(\alpha,\beta) - 2\frac{\beta^{1/2}}{(1+\alpha-\alpha\beta)^{1/2}}\left[1 - (1-F(\alpha,\beta))^{1/2}\right]\right\} \tag{7}$$

where

$$F(\alpha,\beta) = \frac{1+\alpha}{1+\alpha-\alpha\beta}\frac{V_f - V_r}{\phi_B - E_{C\infty} - V_r - kT/e} \tag{8}$$

The various symbols are as follows: ε is the static dielectric constant; ϕ_B is the Schottky barrier height; V_f and V_r are the forward and reverse bias voltages, respectively; $E_{C\infty}$ is the flat conduction band energy, far from the surface; $N_D^{net} = N_D - N_A$; $\alpha = N_T/N_D$; and $\beta = (E_T - E_{C\infty} - kT)/e(\phi_B - E_{C\infty}/e - V_r - kT/e)$. Note that Eq. 4 explicitly contains N_d^{net} evaluated at w_r, and N_T at $(w_r - \lambda)$, a fact that can be important if the layer is inhomogeneous or if a profile of N_T is being generated.

Fig. 3. Effect of a metal Schottky barrier (energy $e\phi_B$) on the surface of GaN. Filled circles denote states occupied by electrons, and empty circles, unoccupied. As demonstrated by the arrows, filled states above the Fermi level E_F (set at $E = 0$) will emit their electrons to the conduction band and this phenomenon results in a capacitance transient. The various symbols are defined in the text.
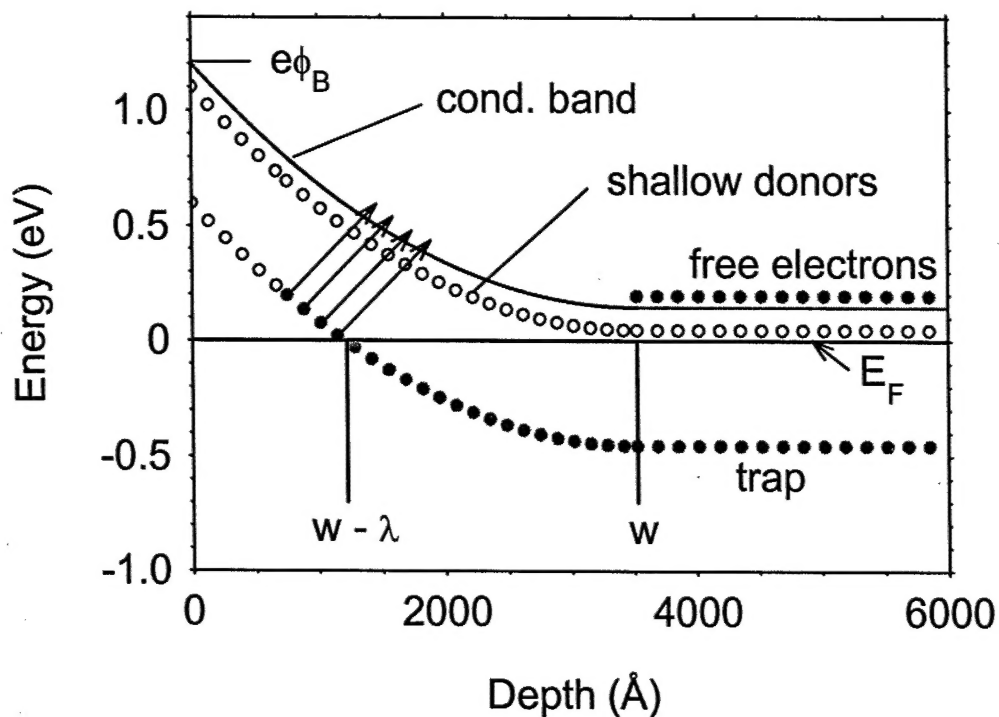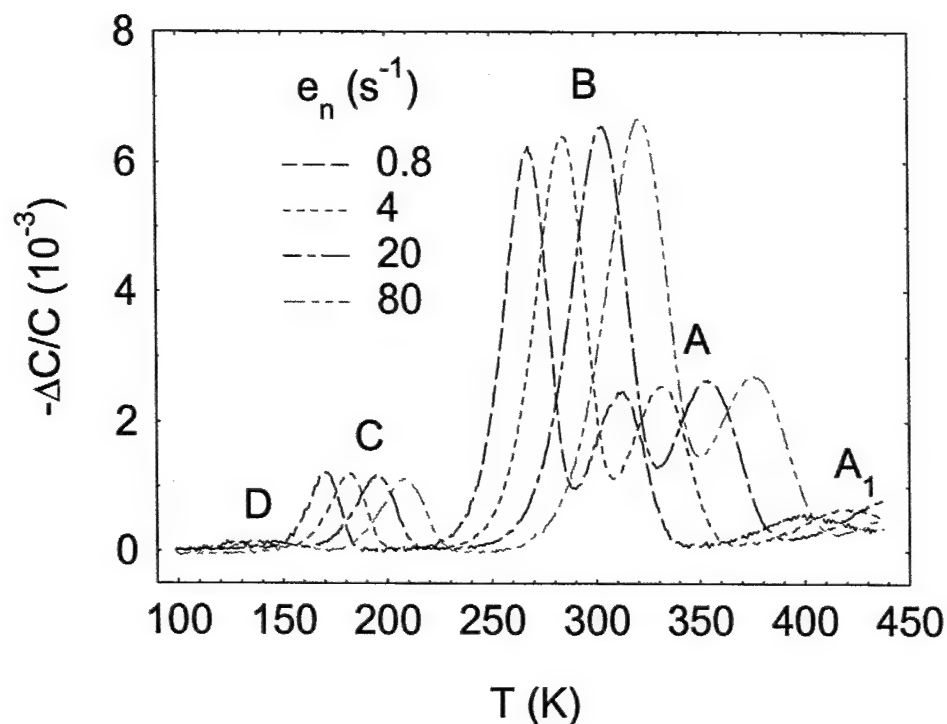
Fig. 4. Plots of -$\Delta C/C$ for a freestanding GaN sample as a function of emission rate e_n. Five traps are identified: A_1, A, B, C, and D. The conditions are: $V_r = -5.0$ V, $V_f = 0$ V, and $t_p = 1$ ms.

In GaN, two common defect-related traps, designated here as ED and AD, are nearly always observed, no matter what type of irradiation is used. For the rate window set at $r = 4$ s^{-1}, traps ED and AD have DLTS signal peaks at about 100 and 400 K, respectively. Other traps have also been seen, as outlined in Table 1, but virtually all workers have at least seen ED and AD. Trap ED was first reported by Fang et al. [15] to have an energy E of 0.18 eV, but later analysis by Polenta et al. [16] revealed that it consisted of two overlapping traps, each with a thermal energy component E_T of 0.06 eV, and capture-cross-section components E_σ of 0 and 0.05 eV, respectively. Indeed, Goodman et al. [17] have recently found that ED consists of three components, but their source of electrons (0.25 – 2.5 MeV, ^{90}Sr) was different from that used by Fang, Polenta, et al. (1-MeV, van de Graaff). The combined production rate of all of the components is about 1 cm^{-1} [17], the same as that determined from the T-Hall analysis [1], and since the thermal energies are also the same (0.06 eV), it is almost certain that a common defect (probably V_N) is being observed. The

different DLTS trap components could perhaps correspond to different separations of the Frenkel-pair components, V_N and N_I [18]. The existence of several components in ED also can explain the slight differences in overall peak position, 0.13 – 0.20 eV, seen by various workers.

It is important to emphasize that the various components of trap ED appear not only in irradiated samples, but also in as-grown, ion-implanted, and contact-metal-deposited samples. In MBE layers [19,20,21], the concentration of trap E_1, one of the components of E_D, increases with decrease of N flux during growth, again supporting its identification with V_N. A strong increase of the trap E_1 DLTS signal with pulse length is illustrated in Fig. 5. This shows that it has a small capture cross section, perhaps due to a repulsive barrier. Incidentally, trap D also must have a defect nature, because we have recently shown that it can be created with plasma etching.

Positron annihilation spectroscopy (PAS)

Positrons injected into defect-free GaN are annihilated by the core atomic electrons in a mean time of 160 – 165 ps. However, if there are negatively charged vacancies present, some of the positrons will become trapped at those locations, and will have longer lifetimes, because of the reduced electron density at vacancies. In the case of GaN, Ga vacancies (but not N vacancies) would be expected to fill this role, and indeed, PAS has been used to identify and quantify V_{Ga}-related defects [22]. For example, it has been shown that 2-MeV electrons produce V_{Ga} centers at a rate of about 1 cm^{-1} in bulk, semi-insulating GaN [11]. Moreover, comparisons of V_{Ga} concentrations with acceptor concentrations N_A in a series of undoped, n-type HVPE GaN samples, with N_A ranging from 10^{15} to 10^{19} cm^{-3}, show that $[V_{Ga}] \approx N_A$, to within experimental error [3,11]. Thus, it appears that V_{Ga}, and not any impurity, is the dominant acceptor in HVPE GaN, and probably in other types of undoped GaN, also. Indeed, theory predicts that V_{Ga} centers should be abundant in n-type GaN[23].

Fig. 5. DLTS signal $-2N_D(\Delta C/C)$ for a MBE GaN layer. Note the strong dependence of trap E_1 on forward-bias pulse length t_p. The conditions are: $e_n = 10$ s^{-1}, $V_r = -3.0$ V, and $V_f = 0.5$ V.
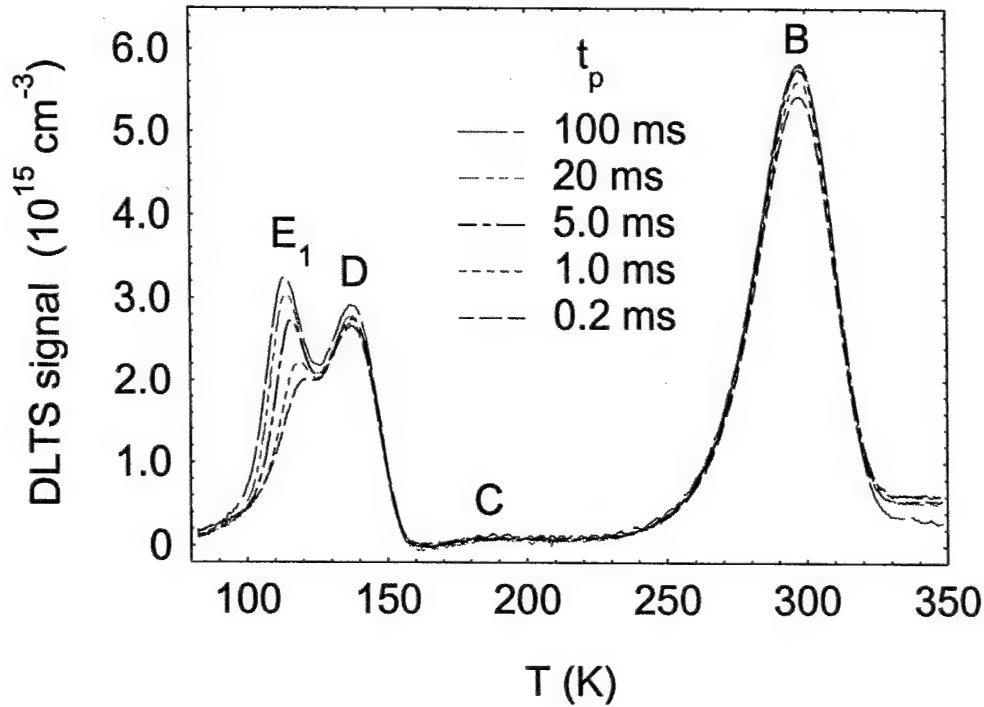
Photoluminescence (PL)

Two, infrared PL bands, at roughly 0.85 and 0.93 eV, are produced by 2.5-MeV electrons [24-27]. Both bands are broad, but the former has a sharp zero-phonon line (ZPL) at 0.88 eV and accompanying phonon structure. Some workers have proposed that the lower-energy band with the ZPL is much like the well-known O_P band in GaP, and, in the GaN case, involves a transition between a deep ground state of O_N (at $E_C - 0.90$ eV) and an excited state of O_N (at $E_C - 0.02$ eV), along with associated phonon side bands [27]. However, there are several apparent problems with this model, and another defect, an O_N-Ga_I complex, may be a better candidate [28].

Dislocation Studies

Recently, threading-edge dislocations (TEDs) have been shown to be negatively charged in n-type GaN, behaving as a line charge with a linear charge density of about 1e per c-lattice distance, 5.185 Å [29]. Thus, these dislocations are acceptor-like, and theoretical studies are consistent with this picture, suggesting that the dislocation cores in n-type GaN may contain Ga vacancies [30,31], or V_{Ga}-O_n complexes, with $n = 1 - 3$ [32]. It might be assumed that simple V_{Ga} centers along the core would have a charge of -3 each, but it turns out that electron-electron repulsion reduces the charge to about -1 each, for typical material with $n \sim 1 \times 10^{17}$ cm^{-3} [31]. For HVPE GaN on Al_2O_3, the interface region has a very high TED density, $N_{dis} \sim 10^{10} - 10^{11}$ cm^{-2}, and an even higher misfit dislocation density, $N_{mis} > 10^{12}$ cm^{-2}, as deduced by transmission electron microscopy (TEM) measurements [3]. Also, from secondary-ion mass spectroscopy (SIMS) measurements, $[O] > 10^{19}$ cm^{-3} in this region; from PAS, $[V_{Ga}] > 10^{19}$ cm^{-3}; and from Hall-effect and electrochemical C-V (ECV) measurements, $n > 10^{19}$ cm^{-3} [3]. The picture that emerges here is that the donors are O, and the acceptors are V_{Ga}, but the V_{Ga} in this case are probably associated with the dislocations, either as isolated centers along the core, or as V_{Ga}-O complexes. Interestingly, O is the dominant donor only in the interface region, since SIMS and T-Hall data show that Si takes over in the bulk region. However, PAS and T-Hall measurements show that V_{Ga} is the dominant acceptor everywhere, both in the interface and bulk regions [3].

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Appendix I. Participants in Wood-Witt Program (as of Sept, 2002)

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Appendix II. Wood-Witt related publications (since August 2000) involving WSU personnel.

1. Z-Q. Fang, L. Polenta, J.W. Hemsky, and D.C. Look, "Deep Centers in As-Grown and Electron-Irradiated n-GaN", in *2000 International Semiconducting and Insulating Materials Conference*, ed. By C. Jagadish and N.J. Welham (IEEE, Piscataway, NJ, 2000) p. 35. Mar01

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10. A. Saxler, D. C. Look, S. Elhamri, J. Sizelove, D. Cull, W. C. Mitchel, M. Callahan, D. Bliss, L. Bouthillette, Sheng-Qi Wang, C. M. Sung, S. S. Park , and K. Y. Lee, "High Electron Mobility in Free-Standing GaN Substrates", Mater. Res. Soc. Symp. Proc. **639**, G7.2.1 (2001). Oct01

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12. D.C. Look, J.E. Hoelscher, J.L. Brown, and G.D. Via, "Electrical Profiles in GaN/Al$_2$O$_3$ Layers with Conductive Interface Regions", MRS Internet J. Nitride Semicond. Res. **6**, 10 (2001). May01

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25. A.Y. Polyakov, N.B. Smirnov, A.V. Govorkov, Z-Q. Fang, D.C. Look, S.S. Park, and J.H. Han, "Deep electron and hole traps in freestanding n-GaN grown by hydride vapor phase epitaxy", J. Appl. Phys. (in press).